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(54) Title: SPIN DRAW PROCESS OF MAKING PARTIALLY ORIENTATED YARNS FROM POLYTRIMETHYLENE  
TEREPHTHALATE

(57) Abstract: A spin draw process for making partially orientated yarn from polytrimethylene terephthalate comprising the steps of:  
(a) extruding and spinning polytrimethylene terephthalate and forming a monofilament or multifilament yarn therefrom, (b) heating  
the yarn by contacting it with a first pair of godets moving at a temperature to give a yarn temperature above the glass transition  
temperature and less than the cold crystallization temperature of polytrimethylene terephthalate yarn, (c) moving the yarn between  
the first godet pair and a second pair of godets, and (d) winding the yarn at a speed of 1 to 15% less than that of the second godet  
pair and at temperature less than the glass transition temperature of polytrimethylene terephthalate yarn.



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SPIN DRAW PROCESS OF MAKING PARTIALLY ORIENTATED YARNS  
FROM POLYTRIMETHYLENE TEREPHTHALATE

Field of the Invention

The present invention relates to the production of partially orientated yarns from polyesters, specifically polytrimethylene terephthalate. More specifically, the present invention is a spin draw process for making partially orientated yarn from polytrimethylene terephthalate.

Background of the Invention

Partially orientated yarn (POY) is filament yarn in which the draw ratio is less than normal, i.e. less than that used to make fully orientated yarn (FOY), resulting in only partial longitudinal orientation of the polymer molecules. This necessary orientation can be created by normal drawing between godets or just by adjusting the speed of the first godet pair relative to that of the spinneret. Generally, in making POY from polyesters, especially polyethylene terephthalate (PET), the extruded or formed yarn goes through a free drop from a spinneret to a winder. This is the most economical process and it works well for PET because PET has a wide processing window, i.e. a winding speed of from 50 to 10,000 metres per minute. This type of process does not work well for polytrimethylene terephthalate (PTT) because it has a much narrower processing window, i.e. 500 to 4000 metres per minute. If the speed is higher than this, then the  $T_g$  of the polymer increases. This makes it hard to establish stable conditions during the drawing process because the yarn is so sensitive to tension when it is formed from PTT.

Spin draw processes are generally used to make FOY. They generally comprise two or three pairs of godets which are generally heated to various degrees and between which the drawing of the yarn takes place. This type of process is not used to make POY because it is more expensive than the process described in the preceding paragraph.

#### Summary of the Invention

In accordance with the present invention there is provided a spin draw process for making partially orientated yarn from polytrimethylene terephthalate comprising the steps of:

(a) extruding and spinning polytrimethylene terephthalate and forming a monofilament or multifilament yarn therefrom,

(b) heating the yarn by contacting it with a first pair of godets at a temperature to give a yarn temperature above the glass transition temperature and less than the cold crystallization temperature of polytrimethylene terephthalate yarn,

(c) moving the yarn between the first godet pair and a second pair of godets, and

(d) winding the yarn at a speed of 1 to 15% less than that of the second godet pair and at temperature less than the glass transition temperature of polytrimethylene terephthalate yarn.

In one embodiment, in (b), the first pair of godets is moving at 1800 to 3500 metres per minute, and in (c), the second pair of godets is moving at 1250 to 4550 metres per minute and is at a temperature to give a yarn temperature of 45 to 120°C, at a draw ratio of 0.7 to 1.3. Preferably, the speed of the first godet pair is from 2000 to 3500 metres per minute. Preferably, the speed of the second pair of godets is from 2450 to 4550 metres per minute, more preferably from 2560 to 4550

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metres per minute. Preferably, the yarn temperature in (c) is from 45 to 100°C, more preferably from 60 to less than 100°C. Alternatively, the yarn temperature in (c) is from 60 to 120°C. Preferably, the draw ratio is 0.7 to less than 1.3, more preferably 0.95 to 1.28.

In a second embodiment, in (b), the first pair of godets is moving at greater than 3500 metres per minute, and in (c), the second pair of godets is moving at 2450 to 10,000 metres per minute and is at a temperature to give a yarn temperature of 80 to 180°C, at a draw ratio of 0.7 to 3.0. Preferably, the speed of the second pair of godets is from 2560 to 10,000 metres per minute. Preferably the draw ratio is from 0.7 to less than 3.0, more preferably from 0.95 to 2.0.

In accordance with the present invention there is also provided a spin draw process for making partially orientated yarn from polytrimethylene terephthalate comprising the steps of:

(a) extruding and spinning polytrimethylene terephthalate and forming a monofilament or multifilament yarn therefrom,

(b) contacting the yarn with a first pair of godets at room temperature,

(c) moving the yarn between the first godet pair and a second pair of godets at a temperature to give a yarn temperature above the glass transition temperature and less than the cold crystallization temperature of polytrimethylene terephthalate yarn,

(d) moving the yarn between the second godet pair and a third pair of godets, and

(e) winding the yarn at a speed of 1 to 15% less than that of the third godet pair and at temperature less than the glass transition temperature of polytrimethylene terephthalate yarn.

In a first embodiment, in (c), the second pair of godets is moving at 1800 to 3500 metres per minute, at a draw ratio of 0.3 to 1.0, and in (d), the third pair of godets is moving at 1250 to 4550 metres per minute and is at a temperature to give a yarn temperature of 45 to 120°C, at a draw ratio of 0.7 to 1.3. Preferably, the speed of the third pair of godets is from 2450 to 4550 metres per minute, more preferably from 2560 to 4550 metres per minute. Preferably, the yarn temperature in (d) is from 45 to 100°C, more preferably from 60 to less than 100°C. Preferably, the speed of the second godet pair is from 2000 to 3500 metres per minute. Preferably, the draw ratio in (d) is 0.7 to less than 1.3, more preferably 0.95 to 1.28. Preferably the draw ratio in (c) is 0.3 to 1.0, more preferably 0.7 to 1.0, particularly 0.95 to 1.0.

In a second embodiment, in (c), the second pair of godets is moving at 1800 to 3500 metres per minute, at a draw ratio of above 1.0 up to 1.05, and in (d), the third pair of godets is moving at 1250 to 4550 metres per minute and is at a temperature to give a yarn temperature of 45 to 120°C, at a draw ratio of 0.7 to 1.3. Preferably, the speed of the third pair of godets is from 2450 to 4550 metre per minute, more preferably from 2560 to 4550 metre per minute. Preferably, the yarn temperature in (d) is from 45 to 100°C, more preferably from 60 to less than 100°C. Preferably, the speed of the second godet pair is from 2000 to 3500 metre per minute. Preferably the draw ratio in (d) is 0.7 to less than 1.3, more preferably 0.95 to 1.28.

In a third embodiment, in (c), the second pair of godets is moving at greater than 3500 metres per minute, at a draw ratio of 0.3 to 1.0, and in (d), the third pair of godets is moving at 2450 to 10,000 metres per minute and is at a temperature to give a yarn temperature of 80

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to 180°C, at a draw ratio of 0.7 to 3.0. Preferably, the draw ratio of step (d) is from 0.7 to less than 3.0, more preferably from 0.95 to 2.0. Preferably, the draw ratio in (c) is 0.7 to 1.0, more preferably 0.95 to 1.0.

5 Preferably, the speed of the third pair of godets is from 2560 to 10,000 metres per minute.

In a fourth embodiment, in (c), the second pair of godets is moving at greater than 3500 metres per minute, at a draw ratio of above 1.0 up to 1.05, and in (d), the  
10 third pair of godets is moving at 2450 to 10,000 metres per minute and is at a temperature to give a yarn temperature of 80 to 180°C, at a draw ratio of 0.7 to 3.0. Preferably, the speed of the third pair of godets is from 2560 to 10,000 metres per minute. Preferably,  
15 the draw ratio in (d) is 0.7 to less than 1.3, more preferably 0.95 to 1.28.

In the process utilizing three pairs of godets, the first set of godets is at room temperature, a small draw or no draw is achieved between the first two sets of  
20 godets, the second set of godets is heated and a major draw may occur between the second and the third pairs of godets. (G3). The third pair is also heated to stabilize the final POY package.

#### Detailed Description of the Invention

25 A spin draw process is described herein for making POY from PTT. POY with a range of elongation and tenacity can be produced by varying spinning and drawing conditions (speeds, temperature, etc.). The purpose of using a spin draw machine to make PTT POY is to stabilize  
30 the fibre against shrinkage and to improve package stability and shelf life by developing a sufficiently high crystallinity and relax the stress built during drawing between godets using heated godets prior to winding.

The drawing process generally involves two or more pairs of godets. For example, Barmag, Toray, Murata, Zimmer, and Teijin Seiki spin draw machines may be used in making PTT POY. In this process a first pair of take-up godets (G1) is used to heat the yarn to a yarn temperature above the glass transition temperature ( $T_g$ ) of PTT, generally greater than  $45^\circ\text{C}$ , but lower than the cold crystallization temperature,  $T_{cc}$ , of PTT (generally less than  $60$  to  $65^\circ\text{C}$ ) such that the fibre can be drawn between the first pair (G1) and a second pair of godets (G2). The precise temperature for pair (G1) will depend on the machine used and is determined by the speed and diameter of the godet and the number of wraps on the godet. The second pair of godets (G2) should be at a higher temperature than that of the first set of godets (G1), preferably to give a yarn temperature of  $80$  to  $90^\circ\text{C}$ . Again, the precise temperatures will depend on the speed and the diameter of the godets and the number of wraps on the godets. The yarn moves from the second pair of godets (G2) to the winder. The relative speeds of the godets and winder are:

(G1) less than, equal to, or greater than (G2) greater than or equal to Winder

A preferred process for carrying out the invention utilizes three pairs of godets. The first pair of godets (G1) is at room temperature, the second (G2) is heated and achieves a small draw, or no draw, and the third (G3) puts the majority of the draw (if there is to be any drawing at all between the godet pairs) into the yarn with drawing between the second (G2) and third godet pair (G3). This third godet pair (G3) is heated to stabilize the final POY package. In this case, the relative speeds of godets and winder are:

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(G1) less than or equal to (G2) less than, equal to, or greater than (G3) greater than or equal to Winder

In this situation where there is to be no draw, the draw ratio between the first two pairs of godets (the ratio of speed of the second godet pair to the first godet pair) should be from 0.3, preferably 0.7, most preferably 0.95, to 1.0 and the temperature of the second pair of godets should be set to give a yarn temperature of PTT above the glass transition temperature ( $T_g$ ) and below the cold crystallization temperature ( $T_{cc}$ ) of PTT yarn. If a small draw between the first two pairs of godets is desired, then the draw ratio may be increased up to 1.05. The draw ratio between the second and third pair of godets should be from 0.7 to 1.3, preferably 0.95 to 1.28, and the temperature of the third pair of godets should be set to give a yarn temperature of PTT the same as the temperature of second pair of godets in the previous embodiment depending upon the speed of the godets as discussed above.

$T_g$  and  $T_{cc}$  can be measured with a differential calorimetre (DSC). The procedure normally used with DSC is heating the yarn at 20 °C/min. and making a temperature scan from -50 °C to 260 °C. The change in endothermic or exothermic response of the yarn is recorded.  $T_g$  is the temperature at which the yarn begins the endothermic change from glassy state to rubbery state (observed by the deflection of the line from the temperature scan of the calorimetre).  $T_{cc}$  is the temperature when the exothermic change from the supercooling of the yarn due to cold crystallization becomes maximal (i.e. at the peak of the scan). The yarn specimen for testing can be obtained from the yarns produced with the speed of the first set of godet pair



set at the desired speed and the yarns wound on the first set of godet pair.

5 In another embodiment, the godet pairs used in the present invention can be a godet and a follower instead of two godets. Oil mist would be used for slow speed and air bearing for speeds greater than 2000 metres per minute.

10 When the speed of the first pair of godets (or the second pair in the embodiment where three pairs of godets are used) is 1800, preferably 2000, to 3500 metres per minute, the draw ratio should be from 0.7 to 1.3, preferably less than 1.3, most preferably 0.95 to 1.28, because sufficient bulk (or crimp) can be generated using a draw texturing machine or any of other types. This  
15 necessary orientation can be created by normal drawing between godets or just by adjusting the speed of the first godet pair relative to that of the spinneret. The temperature of the first pair of godets is set to give a yarn temperature of PTT above the glass transition  
20 temperature, 45°C plus or minus 5 to 10°C, and below the cold crystallization temperature, 60-65°C. The speed of the second pair of godets is 1250, preferably 2450, most preferably 2560, to 4550 metres per minute and the temperature of the second pair of godets is set to give a  
25 yarn temperature of PTT from 45, preferably 60, to 120°C, preferably 100°C and most preferably less than 100°C. This produces a yarn with an elongation of greater than 60 percent and a tenacity of less than 3.0 g/d.

30 When the speed of the first pair of godets is greater than 3500 metres per minute, then the draw ratio should be from 0.7 to 3.0, preferably less than 3.0, most preferably 0.95 to 2.0. The temperature of the first pair of godets is the same as above but the temperature of the second pair of godets is set to give a yarn  
35 temperature of PTT ranges from 80 to 180°C. The speed of

the second pair of godets is 2450, preferably 2560, to 10,000 metres per minute. This produces a yarn with an elongation of greater than 20 percent and a tenacity of less than 5.0 g/d.

5 An example of typical operating conditions is shown below:

Table 1

	Set to give yarn Temperature (°C)	Speed (metres per minute)	Yarn Wraps
1 <sup>st</sup> godet pair	50-55	1800-2500	6-9
2 <sup>nd</sup> godet pair	80-90	2800-3800	5-7
Winder	Room	2800-3600	Yarn tension 0.03 g/Denier tension)

10 The speed (draw) ratio is speed of 2<sup>nd</sup> godet pair / speed of 1<sup>st</sup> godet pair = 1.2-1.7 and speed of 2<sup>nd</sup> godet pair/speed of winder = 1.01-1.10.

15 The key points in selecting the exact conditions to use on a particular machine and with a particular yarn are as follows. The temperature of the first pair of godets must be set to give a yarn temperature of PTT lower than the cold crystallization temperature of Tcc and greater than its glass transition temperature, Tg. The temperature of the first pair of godets is chosen depending on the diameter of the godet, the number of wraps, and the speed of the godet pair. The draw point of the filaments is controlled at the point of the last wrap just before leaving any godet pair. The speed of godet pair is chosen such that the yarn wraps are stable. The temperature of the next pair of godets develops the yarn morphology and stress relaxation such that it is stable against shrinkage. It also controls the yarn boiling water shrinkage. The relative speeds of the

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godet pairs, i.e. the draw ratio, control the elongation and the tenacity of the filaments.

The primary purpose of the process of the present invention is to prevent shrinkage of the POY package during winding and a stable package for long shelf life.

The present invention will now be described by reference to the following Examples:

#### EXAMPLES

##### Spinning for POY

Polytrimethylene terephthalate chips used have 0.92 IV (grade CP509210) for all the Examples 1 to 8. Chips were first dried in the dryer at a selected temperature and period of time to achieve the water content of less than 30 ppm. Chips were then automatically fed to a hopper connected to the extruder and extruded at a selected screw speed and set of zone temperatures. Through the extruder, the molten PTT was then transferred through a filter to a spin beam with a gear pump. Next, the molten PTT then passed through a spinneret with a pre-selected number of holes for desired yarn counts, e.g. 48, 36 or 24 as indicated in column 2 of Table 2. Thereafter, the molten continuous strings were passed through a quench cabinet at a preset draft or air flow e.g. 0.4-0.5 metre/sec. as indicated below, cooled for example at 29°C, and solidified into continuous filaments. The solid continuous filaments (or fibres) were then wrapped around the 1<sup>st</sup> set of heated godets (G1) with a predetermined number of wraps as indicated in Table 2. At a location between the quench cabinet and the 1<sup>st</sup> set of godets, a spin finish applicator was used to apply the oil to the filaments. Next, continuously, the filaments proceeded to the 2<sup>nd</sup> set of heated godets again with a predetermined number of wraps (as indicated in Table 2). Thereafter, the filaments were wound on to a bobbin by a winder. When

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the bobbin reached the weight (for example, 14 Kg.), it was automatically doffed off from the winder as a POY package. The extrusion and spinning conditions are described as follows:

5     For Examples 1 to 6:

      Dryer: 130 °C for 4 hrs, target water (H<sub>2</sub>O) ppm:  
<30, actual: 35 to 45 ppm

      A low temperature heating medium, Dowtherm J  
10     manufactured by Dow Chemical (boiling point of 207 °C),  
      was used.

      Spinning Machine manufactured by Zimmer  
(Germany)/Teijin Seiki (Japan): A commercial spin draw  
machine with 24 positions and 8 ends per position. The  
extruder zone temperature is set at: 245, 250, 255, 255,  
15     260 °C.

      Manifold: 250 °C.

      Spin pack/beam: 255 °C.

      Spin Finish: Takemoto 2471 (manufactured by Takemoto  
Chemical, Inc., Japan) at 0.4% OPU (oil pick up  
20     percentage).

      Extruder (for PET): Single screw of L/D= 24 and 14.85 cm  
in diameter.

      Spinning Extruder Hopper capacity: 5.08 metric tons (5  
tons).

25     Bobbin Tube Size: 112 (inner diameter) x 126 (outer  
diameter x150 mm (stroke length)).

      Gear Pump: 2.4 cc/revolution at 22 revolutions per  
minutes x 4 ports.

      Quench Air temperature/Flow Rate: 29 °C/0.4-0.5  
30     metre/sec.

      The winder was manufactured by Teijin Seiki - Model  
AW912.

      For Examples 1 to 6, neither the heating device for  
the space between the surface of the spinneret and the

beginning of the quench cabinet nor the exhaust system is available in this area. The heating device is normally used to prevent the condensation or crystallization of by-products generated in the extrusion and melt transferring system under heat and released at the exit of the spinneret. An exhaust system, if installed, is to withdraw those by-products.

The conditions for the godets set 1 (G1) and set 2 (G2) and for winder are listed in Table 2, Examples 1 to 6.

Examples 7 and 8:

Dryer: 130 °C for 4 hrs, target water (H<sub>2</sub>O) ppm: <30, actual: 35 to 45 ppm.

Low temperature heating medium (boiling point of 207 °C) was used.

Spinning Machine manufactured by Barmag, Germany: A pilot scale spin draw machine with one (1) position and 6 ends per position. Extruder Zone Temperatures were set at: 240/250/265/255 °C.

Manifold: 250 °C.

Spin pack/beam: 258 °C.

Spin Finish: Lurol PT7087 (manufactured by Goulston Technologies, Inc., USA) at 0.4% OPU (oil pick up percentage).

Extruder: Single screw of L/D= 24 and 14.85 cm in diameter.

Spinning Extruder Hopper capacity: 500 kg.

Bobbin Tube Size: 112 (inner diameter) x 126 (outer diameter x 125 mm (stroke length) and 150 mm tube length.

Gear Pump: 3.0 cc/revolution at 19 revolution/min.

Quench Air temperature/Flow Rate: 29 °C/0.4-0.5 m/sec.

Winder was by Barmag's Craft (Birotor) Winder.

The conditions for the godets set 1 (G1) and, set 2 (G2) and for winder are listed in Table 2, examples 7 and 8.

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The bobbins of POY produced with the above described process and Examples 1 to 8 exhibit yarn properties which are very suitable for further processing into textured yarns with a variety of texturing machines.

5 Fabrics can be made from such yarns for applications in textiles.

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Table 2

Example No.	Yarn Type/Count	Spin Temp. °C	G1/G2 Temp. °C	G1/G2 # wraps	G1/G2 Speed m/min	G2/G1 Speed ratio	Winder Speed m/min	Winder/G2 Speed ratio	Tenacity g/d	Elong. %	BWS %
1	A1. POY 105/48	255	55/80	8.5/5.5	2500/3150	1.26	3050	1.03	3.00	76.2	9.8
2	A2. POY 200/48	255	55/80	8.5/5.5	2500/3150	1.26	3050	1.03	2.78	82.9	7.6
3	A3. POY 105/36	255	55/80	8.5/5.5	2500/3150	1.26	3050	1.03	2.88	81.8	8.2
4	B1. POY 83/24	255	50/80	7.5/4.5	2500/3150	1.26	3050	1.03	3.02	76.2	8
5	B2. POY 85/24	255	50/80	7.5/4.5	2500/3050	1.22	2950	1.03	2.90	84.7	6
6	B3. POY 85/24	255	50/80	7.5/4.5	2915/3600	1.23	3350	1.07	3.31	63.6	10
7	C1. POY 175/48	258	55/90	8.5/5.5	2500/2800	1.12	2700	1.04	2.6	110	?
8	C2. POY 195/48	257	48/80	8.5/5.5	2500/3200	1.28	3230	1.01	3.02	81	10.7

C L A I M S

1. A spin draw process for making partially orientated yarn from polytrimethylene terephthalate comprising the steps of:

(a) extruding and spinning polytrimethylene terephthalate and forming a monofilament or multifilament yarn therefrom,

(b) heating the yarn by contacting it with a first pair of godets at a temperature to give a yarn temperature above the glass transition temperature and less than the cold crystallization temperature of polytrimethylene terephthalate yarn,

(c) moving the yarn between the first godet pair and a second pair of godets, and

(d) winding the yarn at a speed of 1 to 15% less than that of the second godet pair and at temperature less than the glass transition temperature of polytrimethylene terephthalate yarn.

2. A spin draw process according to claim 1 wherein:

in (b), the first pair of godets is moving at 1800 to 3500 metres per minute, and

in (c), the second pair of godets is moving at 1250 to 4550 metres per minute and is at a temperature to give a yarn temperature of 45 to 120°C, at a draw ratio of 0.7 to 1.3.

3. A spin draw process according to claim 1 wherein:

in (b), the first pair of godets is moving at greater than 3500 metres per minute, and

in (c), the second pair of godets is moving at 2450 to 10,000 metres per minute and is at a temperature to give a yarn temperature of 80 to 180°C, at a draw ratio of 0.7 to 3.0.



4. A spin draw process for making partially orientated yarn from polytrimethylene terephthalate comprising the steps of:

5 (a) extruding and spinning polytrimethylene terephthalate and forming a monofilament or multifilament yarn therefrom,

(b) contacting the yarn with a first pair of godets at room temperature,

10 (c) moving the yarn between the first godet pair and a second pair of godets at a temperature to give a yarn temperature above the glass transition temperature and less than the cold crystallization temperature of polytrimethylene terephthalate yarn,

15 (d) moving the yarn between the second godet pair and a third pair of godets, and

(e) winding the yarn at a speed of 1 to 15% less than that of the third godet pair and at temperature less than the glass transition temperature of polytrimethylene terephthalate yarn.

20 5. A spin draw process according to claim 4, wherein:

in (c), the second pair of godets is moving at 1800 to 3500 metres per minute, at a draw ratio of 0.3 to 1.0, and

25 in (d), the third pair of godets is moving at 1250 to 4550 metres per minute and is at a temperature to give a yarn temperature of 45 to 120°C, at a draw ratio of 0.7 to 1.3.

6. A spin draw process according to claim 4, wherein:

30 in (c), the second pair of godets is moving at 1800 to 3500 metres per minute, at a draw ratio of above 1.0 up to 1.05, and

in (d), the third pair of godets is moving at 1250 to 4550 metres per minute and is at a temperature to give a yarn temperature of 45 to 120°C, at a draw ratio of 0.7 to 1.3.

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7. A spin draw process according to claim 4, wherein:

in (c), the second pair of godets is moving at greater than 3500 metres per minute, at a draw ratio of 0.3 to 1.0, and

5 in (d), the third pair of godets is moving at 2450 to 10,000 metres per minute and is at a temperature to give a yarn temperature of 80 to 180°C, at a draw ratio of 0.7 to 3.0.

8. A spin draw process according to claim 4, wherein:

10 in (c), the second pair of godets is moving at greater than 3500 metres per minute, at a draw ratio of above 1.0 up to 1.05, and

15 in (d), the third pair of godets is moving at 2450 to 10,000 metres per minute and is at a temperature to give a yarn temperature of 80 to 180°C, at a draw ratio of 0.7 to 3.0.

## INTERNATIONAL SEARCH REPORT

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A. CLASSIFICATION OF SUBJECT MATTER  
IPC 7 D01F6/62 D01D5/46

According to International Patent Classification (IPC) or to both national classification and IPC

## B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

IPC 7 D01F D01D

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the International search (name of data base and, where practical, search terms used)

EPO-Internal, PAJ, WPI Data

## C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	EP 1 033 422 A (ASAHI CHEMICAL IND) 6 September 2000 (2000-09-06) the whole document	1-8
P,A	PATENT ABSTRACTS OF JAPAN vol. 2000, no. 26, 1 July 2002 (2002-07-01) & JP 2001 254226 A (ASAHI KASEI CORP), 21 September 2001 (2001-09-21) abstract	1-8
P,A	WO 01 66838 A (E.I. DU PONT DE NEMOURS AND COMPANY) 13 September 2001 (2001-09-13) the whole document	1-8
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☒ Further documents are listed in the continuation of box C.☒ Patent family members are listed in annex.

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Date of the actual completion of the international search

7 February 2002

Date of mailing of the international search report

14/02/2002

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# INTERNATIONAL SEARCH REPORT

International Application No  
PCT/EP 01/11608

C.(Continuation) DOCUMENTS CONSIDERED TO BE RELEVANT

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A	<p>EP 0 745 711 A (SHELL INT RESEARCH) 4 December 1996 (1996-12-04) the whole document</p>	1-8

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